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ISOTOPIC ANALYSIS OF FISSION-PRODUCT
IODINE-131 BY NEUTRON ACTIVATION
HENRY T. MILLER
and
RICHARD E. PETERSON

UNITED STATES
NAVAL POSTGRADUATE SCHOOL

THESIS

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IODINE-131 BY NEUTRON ACTIVATION

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Henry T. Miller

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Submitted in partial fulfillment of
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MASTER OF SCIENCE
IN
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Monterey, California

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ABSTRACT

Previous investigations have attempted to determine the isotopic composition of carrier-free Iodine-131 by activation analysis. Gminder and Tyler, and Jones and Crist reported that stable I-127 present was of the order of 100 mg/ml in carrier-free I-131 obtained from Oak Ridge National Laboratory. This was equivalent to approximately 3 mg of I-127 per millicurie of I-131. This amount of I-127 cannot be accounted for on the basis of the fission process alone. Further investigation employing an improved detector for activity measurements gave indication that these results are high by a factor of one hundred or more.

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LIST OF ILLUSTRATIONS

1. Introduction.

Fission-product I-131, a widely used radioisotope, is produced by the Oak Ridge National Laboratory and is represented as carrier-free. (1) The process by which it is produced normally involves the irradiation of U-235 slugs for sixty days at the maximum flux of the ORNL Graphite Reactor. This material is carrier-free in the sense that no iodine is deliberately added to serve as a carrier during chemical separation and purification. However, it has been reported that to achieve a satisfactory yield of I-131, a long period of irradiation is necessary; stable I-127 and long half-life I-129 must be formed in sufficient quantity to serve as carrier for the I-131. (2, 3) Fission-product I-131 cannot be considered, therefore, as truly carrier-free.

The chemical separation of fission-product I-131 has been described by Rupp (3), who stated that after chemical separation and purification, the concentration of I-127 plus I-129 is of the same order of magnitude as that of I-131. Also, Russell (4), using a mass spectrograph, has determined that after the I-131 activity has decayed, the iodine remaining consists of approximately equal quantities of I-127 and I-129. The exact ratios of I-131 concentration to those of I-127 and I-129 will vary depending on irradiation time and decay following irradiation. The ORNL catalog (1) lists the radiochemical purity of fission-product I-131 as greater than 99.9% exclusive of short half-life I-133, and the total solids as less than 2 mg/mc, with concentrations greater than 1 mc/ml. However, the content of total solids has been found to vary. Kahn, Freedman, and Shultz (5) have found that I-131 solutions received from the ORNL usually contain approximately 5 mg/ml total solids.

Gminder and Tyler (6) and Jones and Crist (7), using the same method and similar instrumentation to the present investigation, found that the

1

I-127 concentration was 10^5 or 10^6 times the concentration of I-131. These quantities of I-127 are much higher than expected or than can be accounted for readily. In a single activation, Jones and Crist were unable to demonstrate any activity of I-130 resulting from initial I-129 at the limited flux available.

Thermal neutron activation followed by analysis using gamma-ray spectroscopy comprised the method for determining isotopic composition of carrier-free I-131. In a thermal neutron flux, a fraction of the I-127 becomes I-128 by an (n, γ) reaction. Similarly, I-130 is produced from I-129. For determinations of I-127, irradiation times of five half-lives, or 125 minutes, are sufficient to give 97% of the saturation activity. For this irradiation time, if the quantity of I-129 present is not much greater than that of I-127, activity of I-130 would be negligible. Therefore, for short time irradiations, the measured spectrum of count rate versus energy absorbed in the detector is the sum of the I-128 and I-131 activity. Subtracting the experimentally determined I-131 spectrum yields that due to I-128.

More accurate composition data are needed for fission-product I-131, and if possible, a method of analysis applicable to preparations or relatively large activity. This information would be essential in tracer applications employing a very high specific activity of I-131.

Two variations in activation analysis used previously offer promise of improved accuracy. First, the amount of activated product could be increased. This could be accomplished by increasing the activation flux. Second, the sensitivity could be increased by the use of an improved detector. The present investigation attempted to determine the I-127 content of carrier-free I-131 through the use of an improved detector.

2.   Equipment and Procedure.

The samples of I-131 were prepared for activation by evaporating to dryness a measured volume of solution in a lucite container. (7) The samples were placed at the reactor core center position while the reactor was operating at its nominal flux level of 4.5×10^6 neutrons/cm^2/sec. Irradiation time was five half-lives of I-128, 125 minutes. The flux level was held constant within one percent through any one irradiation, and within five percent for all irradiations. Irradiation times were determined to the nearest minute. Elapsed times between irradiation and start of counting were two to three minutes; measured activities were corrected for decay.

A three inch diameter by three inch thick Harshaw Type 12A12 thallium activated sodium iodide crystal was used for all determinations. This phosphor was mounted on a Dumont Type 6363 photomultiplier tube in a manner similar to that described by Bell. (8) The detector assembly was supported in a lightproof plastic cylinder which, in turn, was surrounded by a 3½ inch inside diameter lucite sample holder. Provision was made for accurately positioning samples at distances of 1, 2, 3, 5, and 10 cm from the face of the crystal. The detector mount and sample holder are shown in Figure 1. Counting was accomplished using a Tracerlab RLP-6 Step Scanning Spectrometer. With this instrument, gamma radiation absorbed by the detector was counted in fifty equal increments over an appropriate selected range of energies. Counting times were accurate to within 0.01 minute.

The resolution achieved with the crystal and associated equipment as usually designated was 7.75%; i. e., for the 0.662 Mev gamma of Cs-137. For energies in the range of interest in this investigation, the resolutions are shown in Table 1 and in Figure 2, plotted as a function of gamma energy.
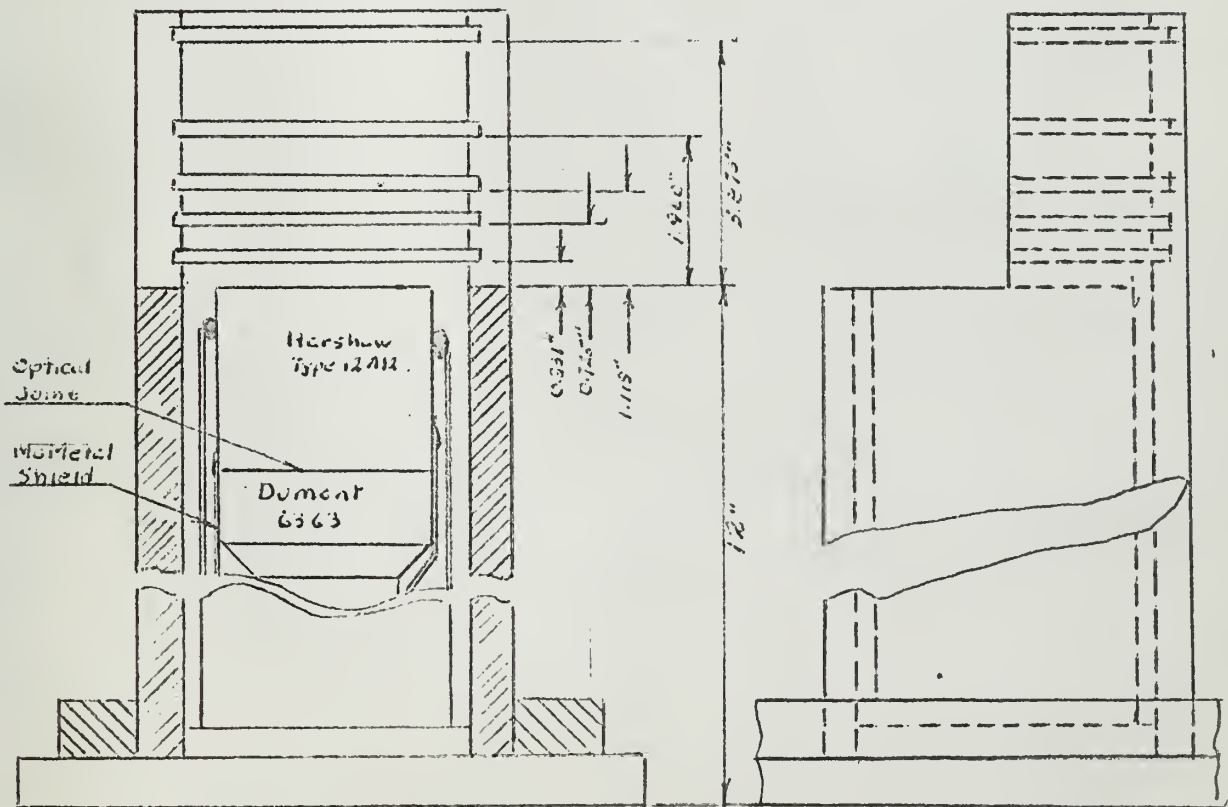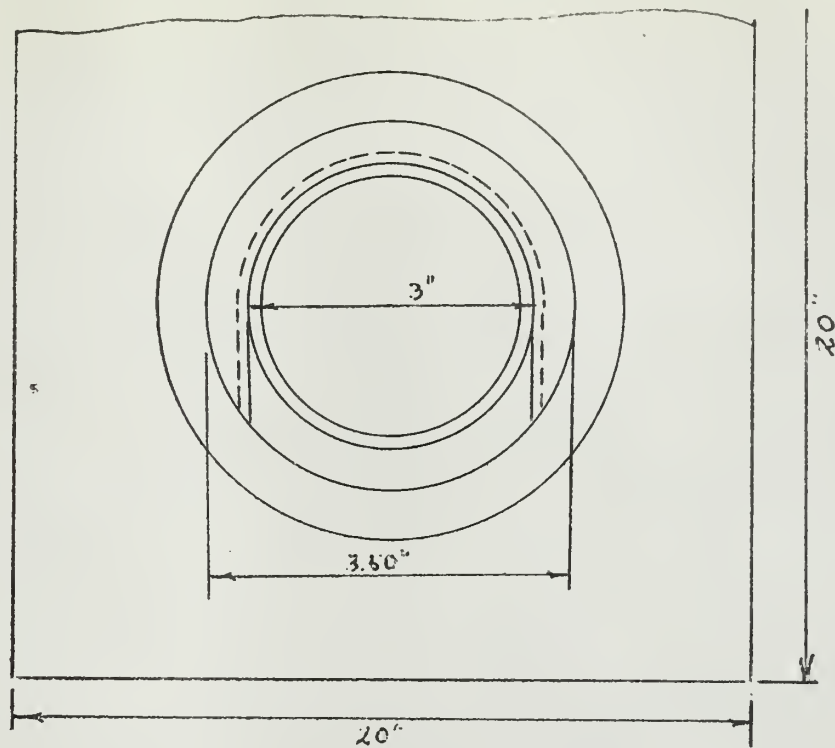
3

FIGURE 1
DETECTOR ASSEMBLY

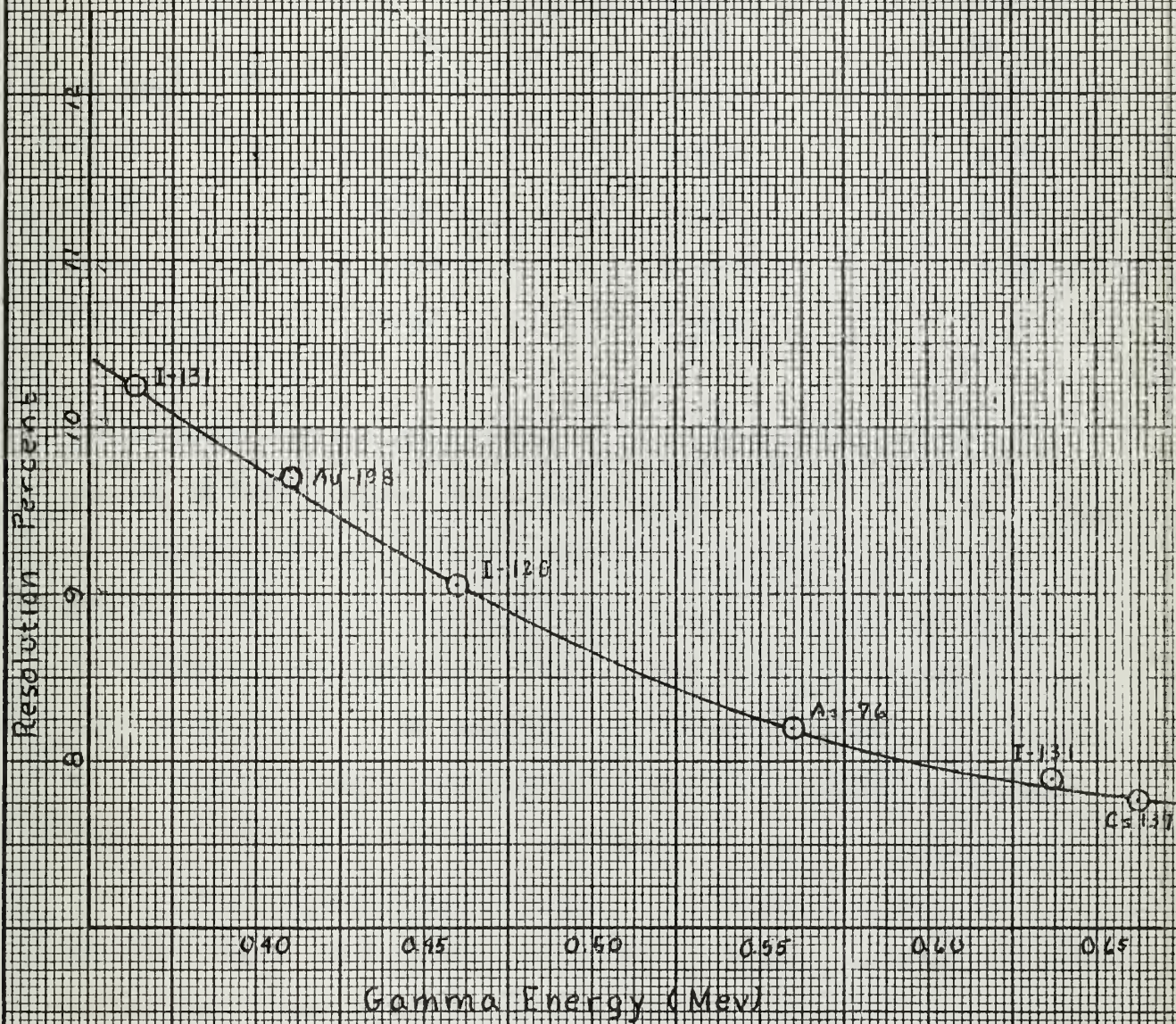FIGURE 2.
CRYSTAL RESOLUTION
3" x 3" Harshaw Type 12A 12

Resolution Percent

Gamma Energy (Mev)

5

Table 1

Isotope	Gamma Energy Mev	Width at Half Maximum Mev	Resolution %
I-131	0.364	0.0373	10.25
Au-198	0.412	0.0399	9.70
I-128	0.46	0.0416	9.05
As-76	0.560	0.0459	8.20
I-131	0.637	0.0503	7.90
Cs-137	0.662	0.0513	7.75

Resolution is defined as the width of the photopeak at one-half of its maximum intensity divided by its gamma energy.

The detector was positioned centrally in a 2 inch thick lined lead shield of inside dimensions 22 x 22 x 24 inches. As shown in Figure 3, the shield was constructed of 2 x 4 x 8 inch lead brick reinforced by a rigid steel frame. It was provided with an inner lining of 0.125 inches of sheet aluminum and an intermediate lining of 0.1875 inches of sheet brass. The greater the distance the shield is removed from source and detector the less backscattered radiation influences the count rate. (9) The contribution due to backscattered and fluorescent radiation was small as is evident from the Cs-137 spectrum shown in Figure 4. This extraneous radiation amounted to four percent of the total measured count rate.

The spectrometer system was calibrated by utilizing appropriate gamma emitters of known energy including I-131, Au-198, Zn-65, As-76, Cs-137, Zr-Nb-95, and Co-60. A substantially linear relationship between quantum energy and pulse height increment was obtained for two different energy ranges. Proportionality for a measurement range from 0 to 1.5 Mev was

26"

26"

28"
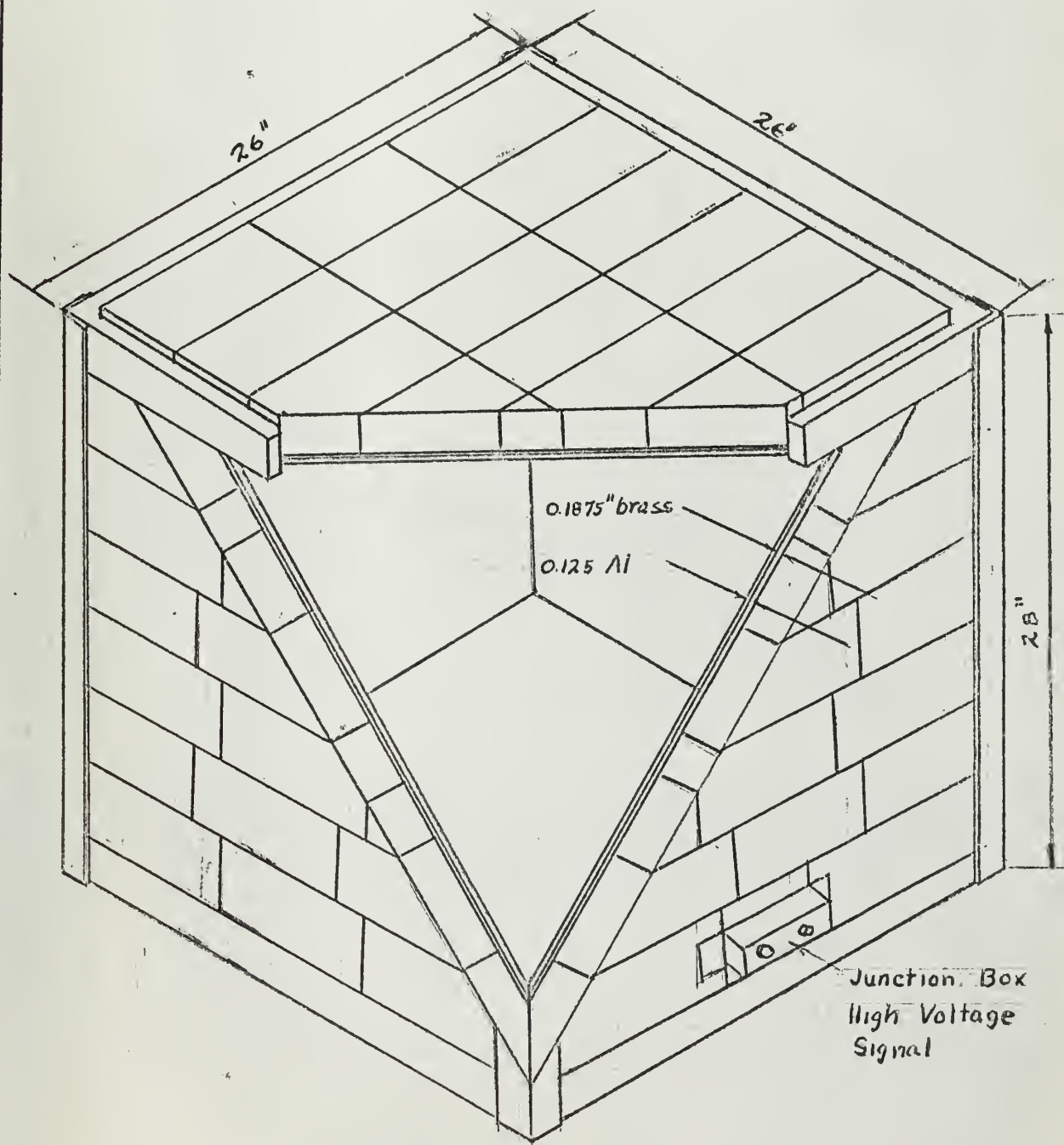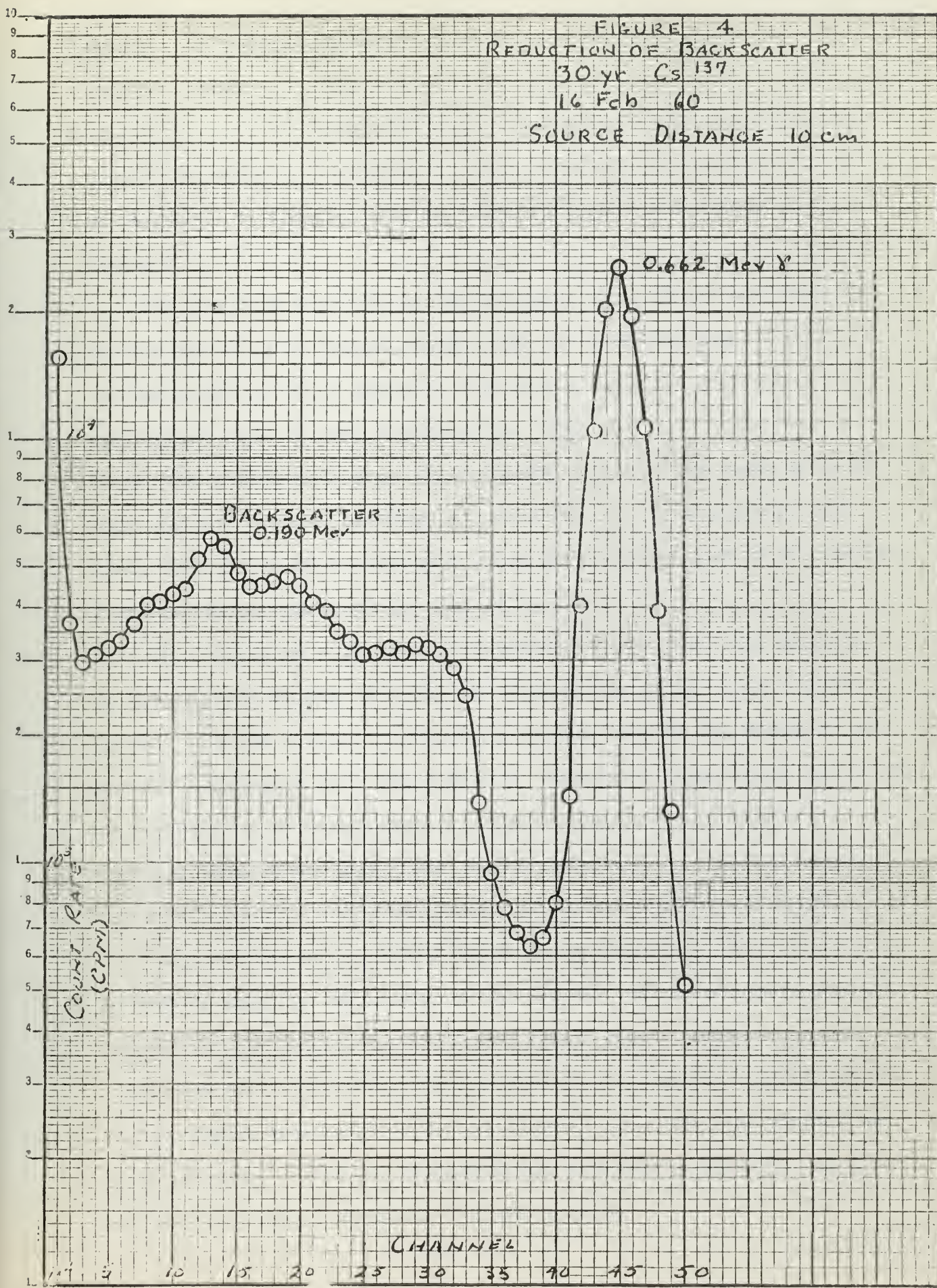
0.1875" brass

0.125 Al

Junction Box
High Voltage
Signal

FIGURE 3
SHIELD CONFIGURATION

FIGURE 4
REDUCTION OF BACKSCATTER
30 yr Cs^137
16 Feb 60
SOURCE DISTANCE 10 cm

0.662 Mev γ

BACKSCATTER
0.190 Mev

10^7

10^6

Count Rate (CPM)

CHANNEL

8

within 1.1% standard deviation from the mean value of energy per channel. Reduction of the energy range covered to 0 to 0.75 Mev gave a variation of 1.9% on the same basis. The latter range of measurements doubled the number of experimental points available in the energy range of interest. Calibration data are shown in Table 2 and plotted in Figure 5.

The long term stability of the equipment observed was such that the maximum variation in channel location for the 0.364 Mev peak of I-131 was approximately three percent. This necessitated the checking of calibration prior to each determination. When the I-131 activity of a sample was sufficiently high, the 0.364 Mev and 0.637 Mev peaks of I-131 were used for internal calibration. When samples of low activity were used, calibration was accomplished using the 0.364 Mev peak of an auxiliary I-131 source and the 0.662 Mev peak of Cs-137.

In a sample of I-131 after activation of I-127, the I-128 spectrum is superimposed on that of I-131 and distorts the latter; with minimum amounts of I-128 activity, it would be evident only in the region of the principal photopeak of I-128, 0.46 Mev. Therefore, counting was restricted to channels in the energy range from approximately 400 to 500 Kev. Counting times of one minute per channel enabled three separate measurements to be made in this energy range within one half-life of I-128. Following determinations of the complex spectrum, the I-128 activity was allowed to decay to one percent of its original value and determination was made of the I-131 spectrum in this energy range. After decay correction of the I-131 spectrum to the time of each original determination of the complex spectrum, subtraction of the I-131 curve then yielded the I-128 activity. Since the background was essentially constant, its effect was cancelled by the subtraction of the spectra.

9

Table 2

CALIBRATION DATA

Isotope	Gamma Energy Kev	For 0 to 1.5 Mev		For 0 to 0.75 Mev	
		Channel	Kev/Channel	Channel	Kev/Channel
I-131	364			25.88	14.06
Au-198	411.8	13.15	31.32	29.25	14.07
Zn-65	510	16.55	30.82	35.80	14.25
As-76	560.5	18.40	30.46	38.85	14.43
Cs-137	661.6	21.67	30.53	44.98	14.71
Zr-Nb-95	751	25.10	29.92		
Zn-65	1119	36.65	30.53		
Co-60	1173	38.10	30.79		
As-76	1210	39.25	30.83		
Co-60	1332	42.55	31.30		
Mean:			30.72 ± .34		14.30 ± .27

FIGURE 5
Spectrometer Calibration

11
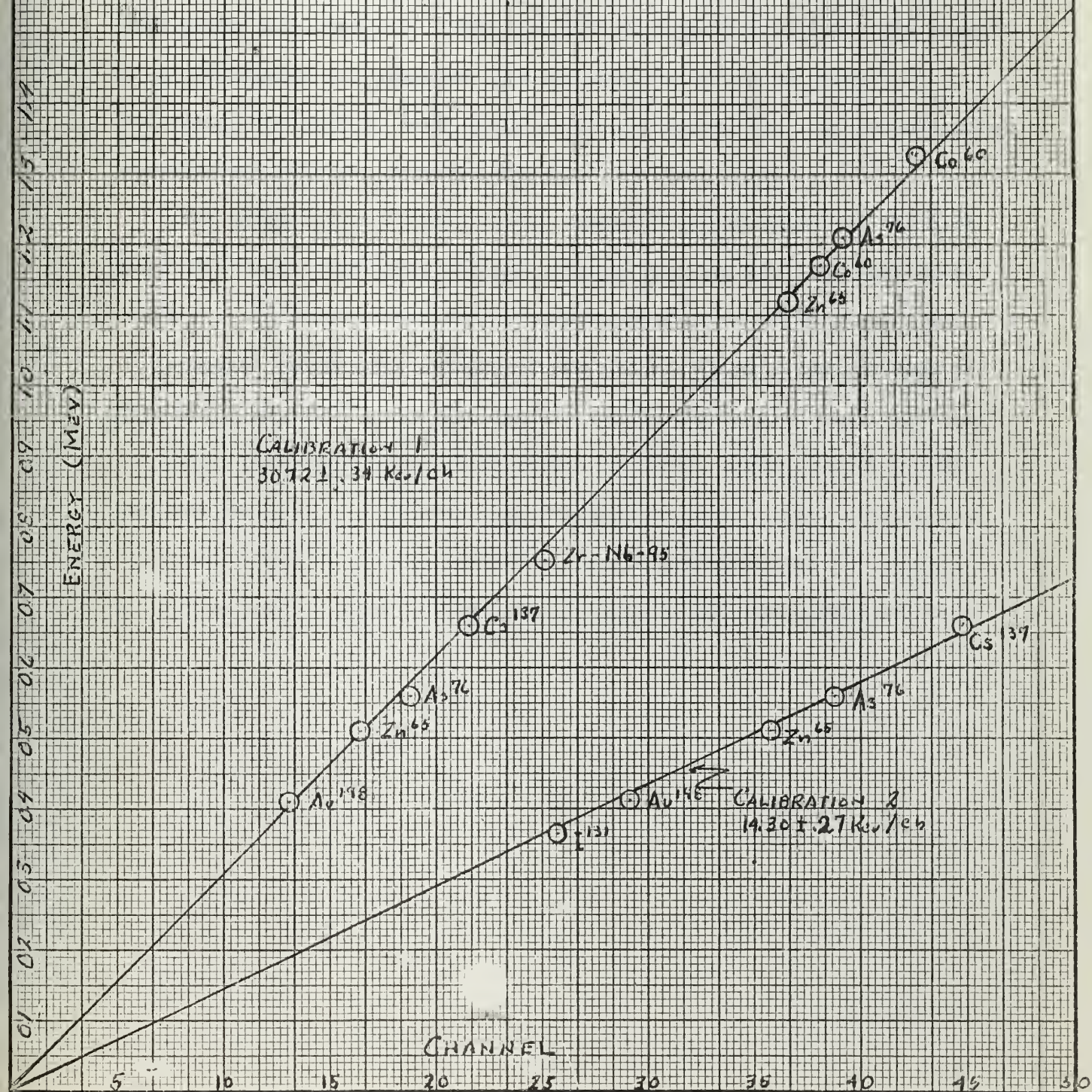
The height of the I-128 photopeak was estimated using the method of C. E. Miller. (10) Idealized photopeaks of various heights and appropriate width for the resolution corresponding to the energy of the I-128 peak were selected. These curves were then fitted to the experimental data with maximum at 0.46 Mev. The value of peak count rate obtained was then decay corrected to the end of irradiation. From the peak count rate, the area under the photopeak was calculated. The amount of I-128 present was computed with appropriate values of detection efficiency and peak to total ratio which are known. (9) The corresponding quantity of I-127 was obtained directly from this result.

The amount of I-127 may also be computed by comparison of the peak count rate with that measured with a known quantity of I-127 activated under the same conditions. (11) A sample, consisting of 36 milligrams of potassium iodide, was irradiated and the resulting I-128 spectrum was determined in order to confirm experimentally the size of the I-128 peak to be expected from a particular amount of I-127. Calculations by the absolute method indicate that this quantity of I-127 should produce peak count rates of 154 cpm at a counting distance of 10 cm and 1110 cpm at 2 cm. The experimental values were 149 cpm and 896 cpm respectively.
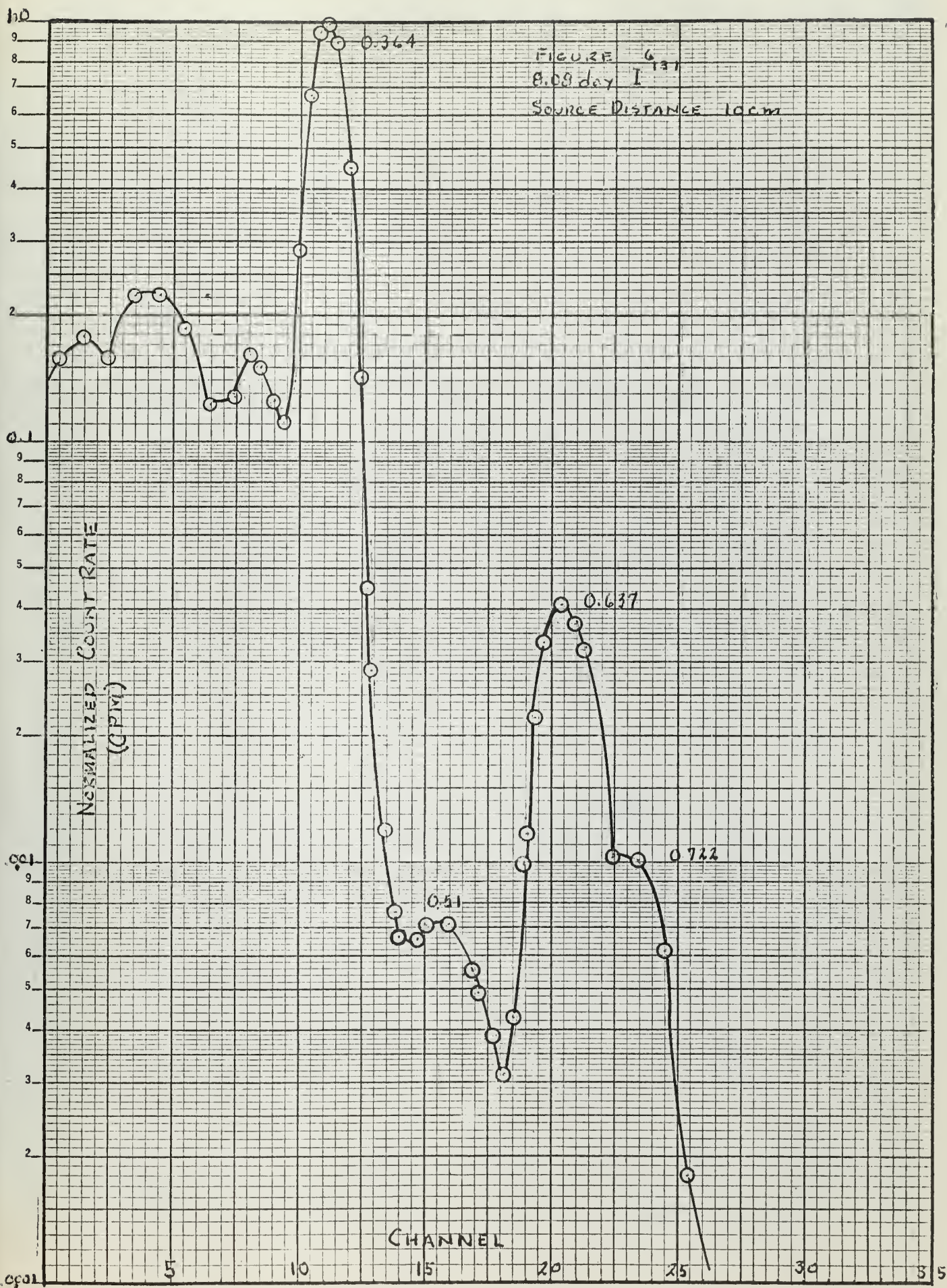
3.   Results.

Experimentally determined I-131 and I-128 spectra are shown in Figures 6 and 7. The I-131 spectrum was obtained using fission-product from ORNL. It represents the mean of eight spectrum determinations normalized to the 0.364 Mev peak for count rate and corrected for drift. The I-128 spectrum was obtained with activated pure crystalline iodine. Three independent determinations in the vicinity of the 0.46 Mev photopeak are shown, one of which covered the entire energy range. This spectrum was normalized also to its principal photopeak.

Four different lots of the carrier-free fission-product I-131 were analized for I-127 content. These lots varied widely in age as did also the quantities of material irradiated.

Four determinations were made using small samples from a lot which had originally assayed 52.9 mc/ml and varied in age from 31 to 46 days. Since the maximum total count rate must be held below 4000 cps to avoid significant coincidence losses in the counting circuitry (6), the sample size and, hence, the amount of material irradiated per determination was limited by the I-131 activity. No I-127 was detected. Since the sample sizes were close to the maximums permissible, it must be concluded that the amount of I-127 was outside the limits of sensitivity, 0.1 mg, as shown in section 4.

Three other lots consisted of much older material. These lots were comparable in age and original assay, and the sample sizes used were 0.2 ml and 1.0 ml. Negative results were obtained in the single determination using Lot 2. One of three determinations involving Lot 3 was positive and gave an I-127 content of 0.1 mg/ml. Three of four results obtained with Lot 4 were positive, with I-127 contents of 0.8, 0.1, and 0.1 mg/ml. A summary of results obtained is shown in Table 3.
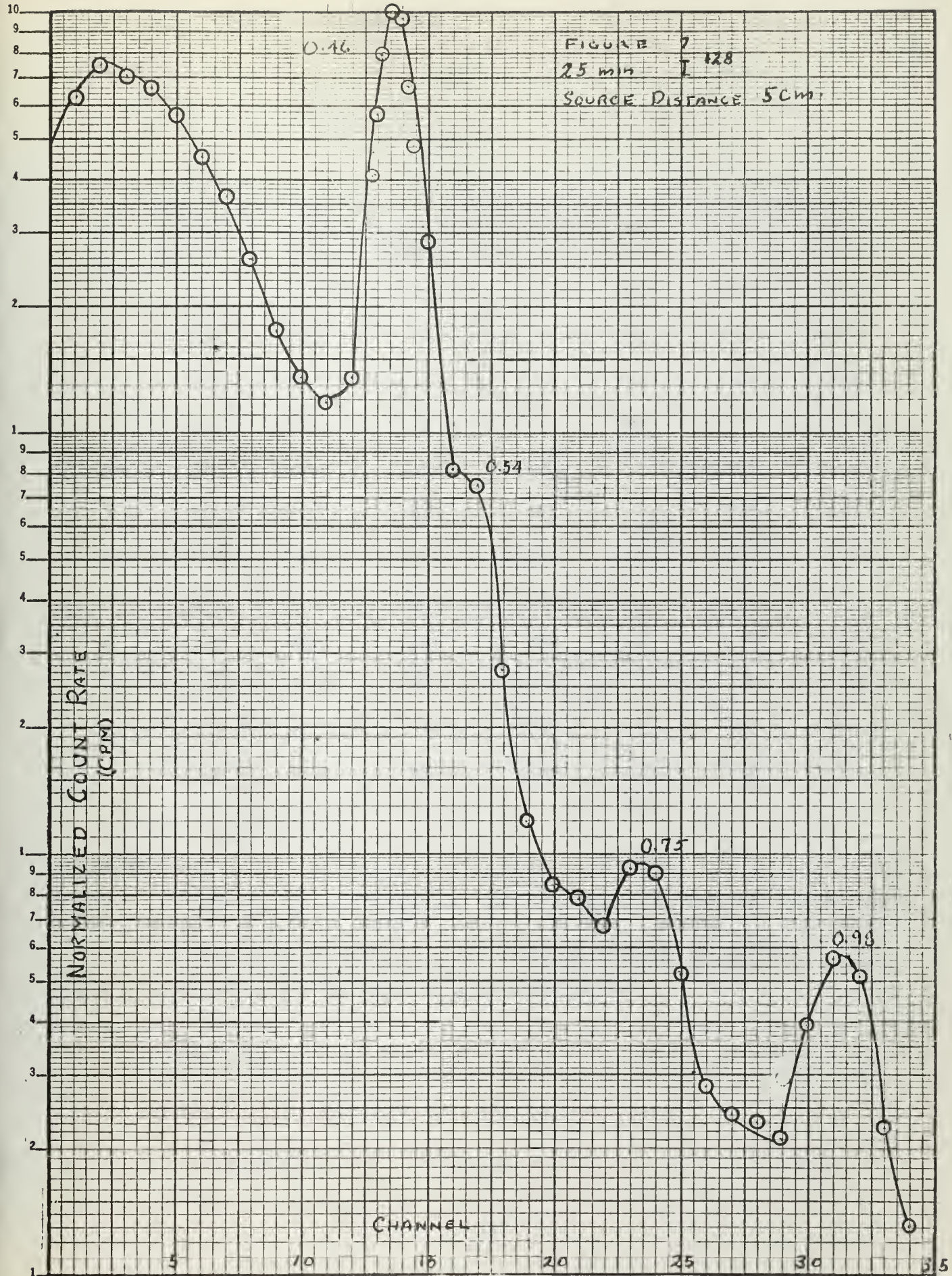
13

FIGURE 6
8.09 day I_{131}
SOURCE DISTANCE 10 cm

0.364

0.637

0.722

0.51

NORMALIZED COUNT RATE
(CPM)

CHANNEL

14

FIGURE 7
25 min   I 128
SOURCE DISTANCE 5 Cm.

0.16

0.54

0.75

0.98

NORMALIZED COUNT RATE (CPM)

CHANNEL

15

Table 3

RESULTS

Lot no.	Determination no.	ORNL assay mc/ml	Age (days)	Quantity irradiated λ	Cm above crystal	Photopeak count rate cpm	I-127 content mg
1	1-1	52.9	31	1	10	~0	<0.1
1	1-2	52.9	35	1	10	~0	<0.1
1	1-3	52.9	41	5	10	~0	<0.1
1	1-4	52.9	46	5	10	~0	<0.1
2	2-1	39.2	409	200	2	~0	<0.1
3	3-1	30.3	207	200	2	~0	<0.1
3	3-2	30.3	210	1000	2	~0	<0.1
3	3-3	30.3	224	1000	2	3	0.1
4	4-1	27.7	344	200	2	5	0.2
4	4-2	27.7	350	1000	2	4	0.1
4	4-3	27.7	354	1000	2	~0	<0.1
4	4-4	27.7	364	1000	2	2	0.1

NOTES:

(1) In determination 4-4, detailed spectrum analysis was not made.

(2) One milligram of I-127 would be expected to produce a peak count rate of 33 cpm at a distance of 2 cm above the crystal, or 5.5 cpm when at a 10 cm distance.

4. Conclusions.

The absolute magnitude of the count rates obtained even with large and old samples was very low. Hence, the accuracy of the peak count rate determination was necessarily poor due to scatter of the experimental points in the vicinity of the peak and may not be statistically significant. It is reasonable to attribute the negative results observed to this uncertainty. Accordingly, the results can be relied upon only in order of magnitude for the quantity of I-127. This appears to be about 0.1 mg/ml and in any case less than 1 mg/ml. The lower of these estimates corresponds to 0.003 mg/mc of original I-131. The detector system of the present study was greatly improved by the use of a larger shield and a larger and superior crystal. Effects due to backscattered and fluorescent radiation were unimportant. The reduction of these radiations was such that there was no distortion of the spectrum in the energy range of principal interest. The crystal resolution of 7.75% was adequate to readily distinguish the principal I-128 peak from peaks due to I-131. The improved sensitivity resulting, however, was not adequate to allow satisfactory estimation of the amount of I-127 present.

With the present equipment and flux, it should be possible to determine a quantity of one milligram of I-127 with an accuracy of 15% on the basis of results obtained with a known quantity of iodine. A non-quantitative indication of the presence of I-127 would be given by one-tenth this amount. Assuming an I-127 content of 1 mg/ml and an assay of 35 mc/ml, it would be necessary to allow decay for 100 days for 15% accuracy of analysis. At much higher specific activities, detection of I-127 would not be expected. The sensitivity of the determination could be increased by increasing the activation flux. Increasing the flux by a factor of 200 would allow detection only, of 5×10^{-4} milligrams of I-127.

Based on previous results (6,7) the I-127 present in a one milliliter sample was about 100 mg. This quantity is well within the sensitivity of the present investigation. When counted at 2 cm, the expected peak count rate would be 3300 cpm which is much greater than any observed. It would appear that differences between present and previous results are attributable either to improved detection equipment or to actual differences in the samples. Since even an I-127 content of 0.1 mg/ml cannot be accounted for on the basis of fission, it appears most likely that contamination during chemical processing must be considered. If this is the case, large variations in I-127 content might be expected.

5. Summary.

(1) The quantity of I-127 present in carrier-free I-131 was no greater than 0.03 mg/mc of original activity. This is lower by a factor of one hundred than the quantities previously reported.

(2) Improved spectral analysis made possible by good crystal resolution, and the reduction of backscattered radiation through shielding design were inadequate alone to permit determination of I-127.

(3) With the present equipment and a flux of 4.5×10^6 neutrons/ cm^2/sec, the minimum quantity of I-127 determinable is of the order of one milligram.

(4) For more satisfactory estimation by activation methods of the isotopic composition of fission-product I-131, a greater neutron flux is essential.

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Determination of a Photopeak

The experimentally determined photopeak is assumed to have a Gaussian distribution about its most probable value. Its semilogarithmic plot is a parabola whose equation is

$$Ln\ y = Ln\ y_o - Ax^2 \qquad \text{where } A = 1/(2\sigma^{-2})$$

and where

y   = Number of counts observed in a particular interval

y_o = Peak count

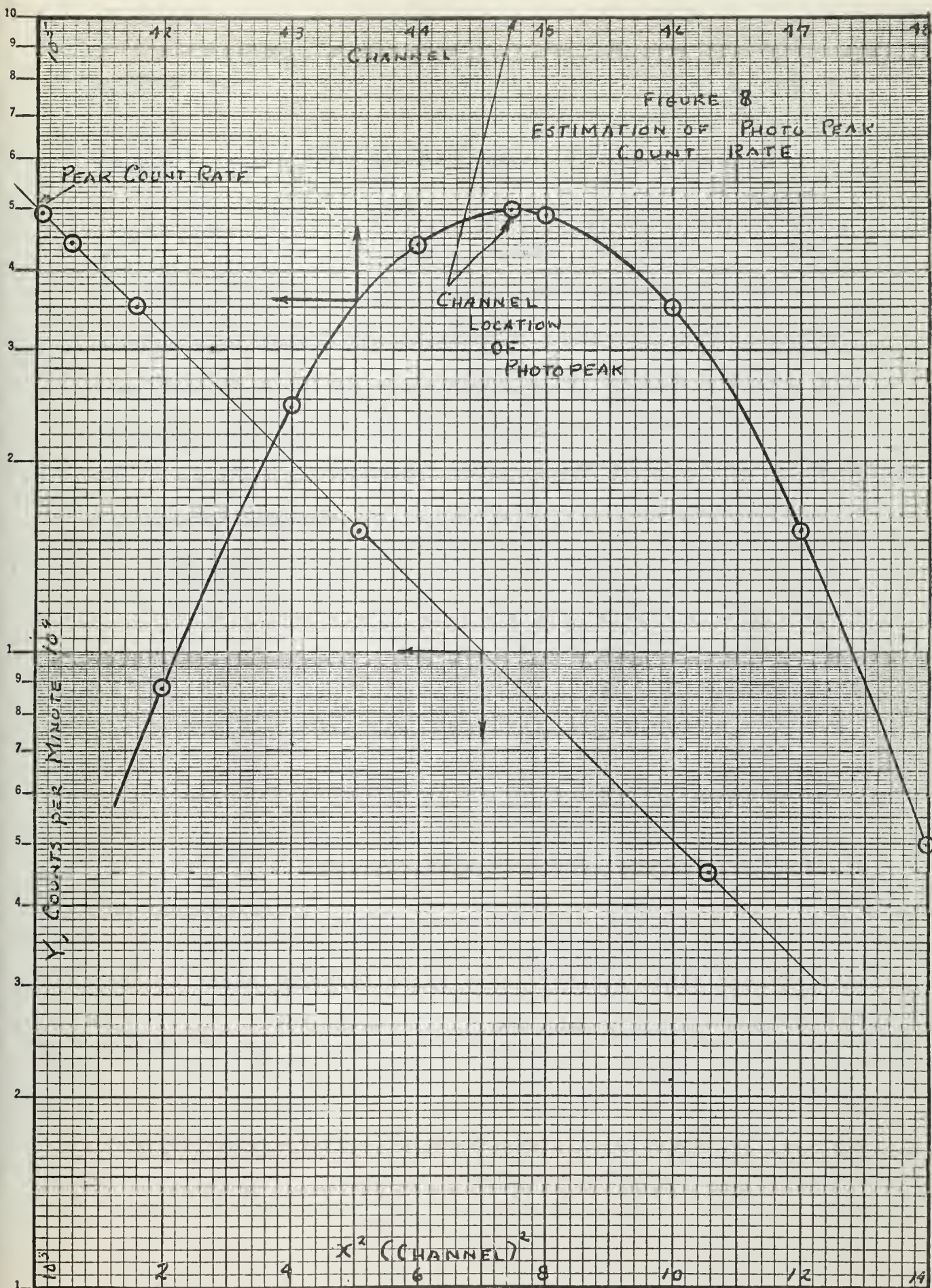A   = Slope of Ln y versus x^2

σ = Standard deviation for the Gaussian distribution

x   = Difference of abscissas of y_o and y in appropriate energy units

In determining the channel (or energy) location of a photopeak, an estimate is made of this location. Then several points in the vicinity of the peak, and on the high energy side, if possible, are selected. Using these points, a plot is made of Ln y versus x^2. If the energy of the peak was estimated correctly, the plot obtained should be a straight line. Otherwise, successive approximations yield a best fit. The y axis intercept yields the peak count and σ may be evaluated from the slope. The area under the photopeak and detector resolution are readily obtained when these parameters are known. An illustration of the technique follows:

The following data are plotted in Figure 8

Channel	44	45	46	47	48
y	44,002	48,990	34,998	15,504	4,490

FIGURE 8
ESTIMATION OF PHOTO PEAK
COUNT RATE

PEAK COUNT RATE

CHANNEL

CHANNEL
LOCATION
OF
PHOTOPEAK

Y, COUNTS PER MINUTE 10⁴

X^2 (CHANNEL)²

23

Assume that the peak is located at channel 44.75 and compute

values of x^2

Channel	44	45	46	47	48
x	0.75	0.25	1.25	2.25	3.25
x^2	0.5625	0.0625	1.5625	5.0625	10.5625

Plot values of x^2 versus Ln y as shown in Figure 8. Since a
straight line results, the assumed peak location is correct. A peak
count rate of 50,000 cpm may be read from the straight line plot; and
from this line, the half width at half maximum is found to be
$(3)^{1/2} \cong 1.732$. The area under the photopeak is calculated as 184,900 cpm.
Finally, the resolution is computed to be

$$\frac{2\,(1.732)}{44.75} = 7.75\%$$

APPENDIX II

Activation Calculations

Part 1 Absolute Method

Symbol	Meaning
y_0	Peak count rate
σ	Standard deviation for a Gaussian Distribution
$W_{\frac{1}{2},\frac{1}{2}}$	Width of Photopeak at half maximum
R	Absolute gamma emission rate
R_p	Gamma emission rate under photopeak
p	Peak-to-total ratio
e_t	Total absolute efficiency of crystal
A	Correction factor for absorption
α	Activity in disintegrations/second
σ_{127}	Activation cross section of I-127
$\emptyset$	Reactor flux in neutrons/cm^2/sec
λ_{128}	Decay constant for I-128
t	Time of irradiation
W_{127}	Mass of I-127 in grams
N_{127}	Number of I-127 atoms in sample
M_{127}	Atomic mass of I-127
0.17	Fraction of decay resulting in 0.46 Mev gamma
6.025×10^{23}	Avogadro's Number

Gamma emission rate under photopeak

$$R_p = \sqrt{2\pi}\,\sigma\, y_0 \qquad \text{or} \qquad R_p = 2.129\, W_{\frac{1}{2},\frac{1}{2}}\, y_0$$

Absolute gamma emission rate

$$R = \frac{R_p}{p\, e_t\, A}$$

25

Activity induced

$$\alpha = \frac{R}{0.17}$$

Number of I-127 atoms in sample

$$N_{127} = \frac{\alpha}{\sigma_{127}\emptyset \ (1 - e^{-\lambda 128 t})}$$

Grams of I-127 in sample

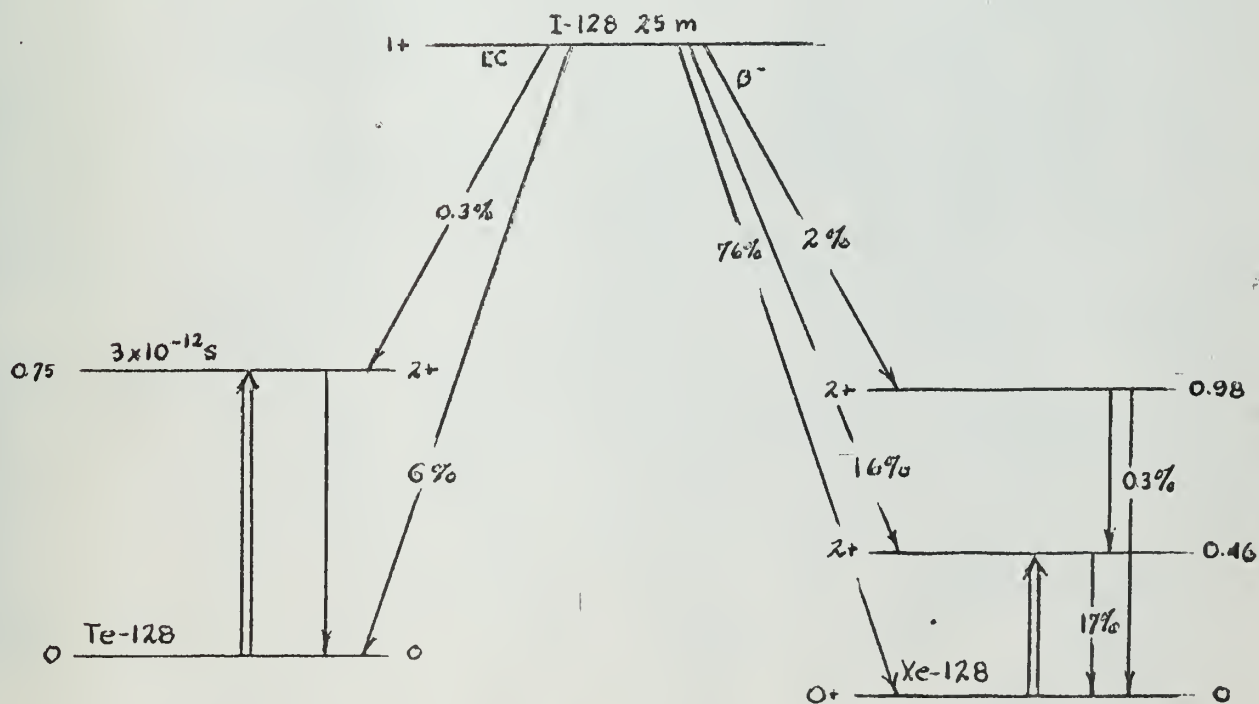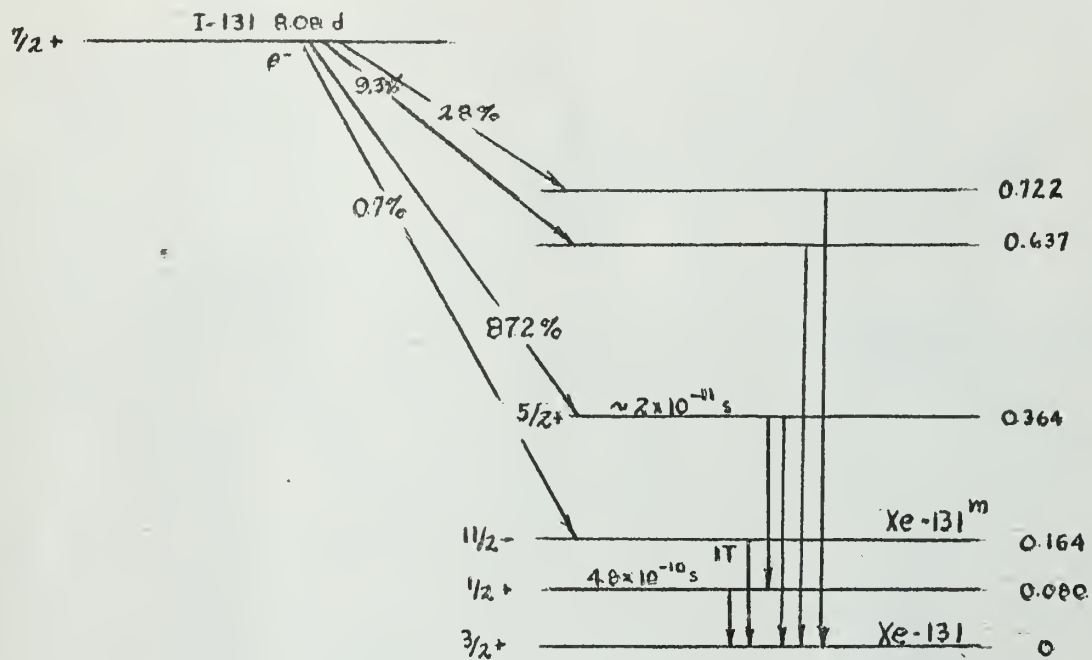$$W_{127} = \frac{N_{127} \ M_{127}}{6.025 \times 10^{23}}$$

Part 2   Comparator Method

Symbol	Meaning
W_u	Mass in unknown in grams
W_c	Mass in comparator in grams
α_u	Activity of unknown
α_c	Activity of comparator

Mass of I-127 in unknown

$$W_u = \frac{\alpha_u \ W_c}{\alpha_c}$$

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I-131- I-128 DECAY SCHEMES